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Woodward-Clyde Consultants

30 October 1990

WCC'D 10-31-90
F.B.


Mr Frank Battaglia
USEPA Region I
Waste Management Division
90 Canal Street
Boston, MA 02114

Dear Frank:

Enclosed are the markup Region I Worksheets. An example Summary Table has been completed for the VOA Appendix IX fraction. Of course, similar tables are currently being generated for the other fractions. A clean finalized Worksheet for both Organic/Inorganic will be forwarded once completed by WCC.

If you have any questions on the above, please do not hesitate to contact me at 201-785-0700, Extension 372. USEPA Region I comments on the worksheets are to be forwarded to Diane Baldi at Ciba-Geigy (919-632-6000).

Very truly yours,


John P. Lorenzo
Project Chemist
Ciba-Geigy

JPL:ef

cc: Mark Houlday (WCC)
Joanna Hall (Alliance)



**OUTLINE OF ERRATA (REMOVAL) AND CHANGES/ADDITIONS
TO THE ORGANIC/INORGANIC
REGION I WORKSHEET RE-EDITED FOR APPENDIX IX ANALYSES**

Errata/Changes/Additions

Organic Region I Worksheet

1. Page 1 of :
 - Removed: Case No. SDG No., Traffic Report Nos. because all information generated is for one data validation source for one site (Ciba-Geigy, Cranston, RI). As such, these sample tracking devices are not required. A Reference No. will be utilized for each data package for WCC/Ciba-Geigy tracking in place of a Case No.
 - Deleted SOW No., Replace with SW-846 (3rd edition).
 - Deleted the partial sentence: and that associated...EMSL-LV, and SMO.
 - Added Sample Identifiers in place for Traffic Report NOS.
2. Page 2 of : No changes/additions warranted.
3. Page 3 of :
 - Added: Waters: Extracted within 7 days, analyzed within 40 days in accordance with Method 8270, SW-846 (3rd edition).
 - Added: Soils (Solids): Extracted within 14 days, analyzed within 40 days.
 - Added: Reference to Table 4-1 of Section 4 of SW-846 and Region I protocols (ie, HT of 7d for VOA aromatics).
4. Page 4 of :
 - Added: BFB and DFTPP tune criteria for CLP and SW-846 are the same.
5. Page 5 of :
 - Added: If any compound has a % RSD > 30% or a % D > 25% for volatiles and > 30% for semi-volatiles.

**OUTLINE OF ERRATA (REMOVAL) AND CHANGES/ADDITIONS
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REGION I WORKSHEET RE-EDITED FOR APPENDIX IX ANALYSES**

6. Pages 6-8 of : No changes/additions warranted.
7. Page 9 of : Surrogate windows entered from Table 5-4 of Radian QAPP.
8. Page 10 of : Delete on Form 3. Added in SW-846, followed by this sentence: Radian Corporation must supply the new SW-846 percent recoveries and RPD maximums on an EPA Form III. Contact Radian Corporation if the revised Form III is not present. Added: MS/MSD RPD maximums and recoveries are listed. Refer to Appendix A. Values obtained from Radian QAPP Table 5-4.
9. Page 11-13 of : No changes/additional were warranted.
10. Page 14 of : Added the note: A megabore or capillary column standard RT may be shorter than 12 minutes.
11. Pages 15-17 of : No changes/additions were warranted.
12. Page 18 of : Deleted the first sentence under analytical sequence. Inserted: Did the laboratory supply the analytical sequence utilized and the appropriate retention time windows for each analyte in the check standard as per the requirements of Method 8000 in SW-846 (3rd edition). After the sentence: Discuss any actions below, added this sentence: Refer to Method 8000 protocols for guidance on actions taken. Contact the laboratory to discuss any anomalies encountered to prevent reoccurrence on future analyses.
13. Page 19 of : Delete < 15%; insert \pm 15%.
14. Page 21 of : Additional Appendix IX insert involving Methods 8080, 8140, and 8150 Surrogate and MS/MSD limits applied from Radian QAPP Table 5-4.
15. Page 24 of : Delete the words Case and SOW, replaced with Reference and SW-846, 3rd edition), respectively.

Appendix A: Matrix Spike RPD Maximums and Percent Recoveries
(VOA-NVOA-Pest/PCB)

Appendix B: Dioxin Summary Forms: Method 8280 (Full Scan: Tetra-OCTA)

**OUTLINE OF ERRATA (REMOVAL) AND CHANGES/ADDITIONS
TO THE ORGANIC/INORGANIC
REGION I WORKSHEET RE-EDITED FOR APPENDIX IX ANALYSES**

Inorganic Region I Worksheet

1. Page 1 of 18 :
 - Deleted the term: Contract Laboratory
 - Replaced with: SW-846 (3rd edition)
 - Deleted Case No. SAS No. and Traffic Report Nos.
 - Replaced Traffic Report Nos. with Sample Identification.
 - Deleted: SOW No. and replaced with SW-846 (3rd edition).
 - Deleted: the partial sentence: and that associated report..., EMSL-LV, and SMO.
2. Page 2-3 of 18 : No changes/additions warranted.
3. Page 4 of 18 :
 - Added: The percent recovery criteria being \pm 10% of the initial value.
4. Page 5 of 18 :
 - 2A: Deleted the term: The SOW; replace with SW-846 (3rd edition).
 - 2B: Deleted the word: analysis. Replace with "day (or every 8 hours), whichever is more frequent".
 - 2C: Between the words were and calibration, inserted the word midpoint. Deleted the partial sentence: or every two hours...frequent. A question mark (?) was placed after ten percent.
 - 2E: Deleted this entire sentence because not a SW-846 (3rd edition) protocol.
5. Page 6 of 18 : Inserted: (A) - (C), three new frequency requirements as per SW-846 (3rd edition) protocols.

**OUTLINE OF ERRATA (REMOVAL) AND CHANGES/ADDITIONS
TO THE ORGANIC/INORGANIC
REGION I WORKSHEET RE-EDITED FOR APPENDIX IX ANALYSES**

6. Page 7 of 18 :
 - Added the note: The SW-846 (3rd edition) requirement is that the calibration blank be within 3 standard deviations of the mean blank value. As such, gross blank contaminations warrants the data validator to contact the laboratory to verify this was performed. List all anomalies in the Inorganic Regional Data Assessment.
 - Deleted: Volume diluted (200 ml). Insert (100 ml) in mg/kg conversion equation.
7. Page 8 of 18 : Added after 1. Recovery Criteria. SW -846 (3rd edition) does warrant a $\pm 20\%$ window of the true value. As such, all Action Percent Recoveries are considered acceptable.
2. Deleted the words sample analysis and insert: batch analysis.
8. Page 9 of 18 : No changes/additions were warranted.
9. Page 10 of 18 : Deleted VI-2B as per SW-846 (3rd edition) protocols do not apply to this criteria.
10. Page 11 of 18 : PQL for tin in a water matrix: Radian PQL implemented - 100 ug/l.
Replace the term CRDL with PQL. Added the footnotes 1-* no detection limits are required in SW-846 (3rd edition). Added MDLs are of a recommended nature. As such, all CLP-CRDLs will be substituted as PQLs for reporting purposes. 2-** Mean RPD of $\pm 25\%$ or $\pm 20\%$ (whichever is tighter) for all analyses greater than 10 x IDL is warranted based on SW-846 (3rd edition) protocols. All Actions are appropriate on this page (#1, #2) except a word change of CRDL \rightarrow PQL.
11. Page 12 of 18 : Same compound changes as page 11. Deleted the metals not part of the Appendix IX analyses: AL, Cd, Fe, Mg, Mn, K, and inserted tin with 100 ug/l PQL obtained from Radian QAPP as the PQL for tin. Convert CRDL to PQL.
12. Page 13 of 18 : Added the note: The SW-846 (3rd edition) LCS recovery window is $\pm 20\%$. The current 80-120% is acceptable. Deleted second

**OUTLINE OF ERRATA (REMOVAL) AND CHANGES/ADDITIONS
TO THE ORGANIC/INORGANIC
REGION I WORKSHEET RE-EDITED FOR APPENDIX IX ANALYSES**

sentence is 2. Solid LCS; insert: Lot specifications are available on request from Radian. Added the word manufacturers after the word EPA.

13. Page 14 of 18 : Deleted 1. Inserted: If duplicate injections do not agree with $\pm 20\%$ for samples/elements, the laboratory must rerun and report the lowest coefficient of variation as per SW-846 (3rd edition) protocols. In 2, edit 85-115% to 75-125% in accordance with SW-846 criteria. Added the note: CLP requirements are not SW-846 (3rd edition) protocols will be used as guidance when applying the qualification actions below.

All references to the Method of Standard Additions were deleted. As part of Round I (chemical indicator selection process from the Appendix IX list), all analytes warranting MSA will be J flagged. Once the indicator list is selected for Round II, MSA will be re-introduced as part of the Round II field sampling program.

14. Page 15 of 18 : After first paragraph, added the statement: as per the CLP guidance and not SW-846 (3rd edition) protocols. Added note: Sample result must be $\geq 50\%$ PQL for calculations by serial dilution; then use $\pm 10\%$ original undiluted value as criteria.
15. Page 16 of 18 : No changes/additions were warranted.
16. Page 17 of 18 : Convert 200 ul to 100 ul; added the word SOLID digestion equation. (2) Radian lab formulas inserted.
17. Page 18 of 18 : Deleted: Case No., inserted Reference No., Delete DPO Action, FYI; Delete SOW, inserted SW-846 (3rd edition). MSA (not performed) was inserted. Refer to #13 above.

REC'D

10-31-90

F.B.

CIBA-GEIGY

ORGANIC REGION I WORKSHEETS

RE-EDITED FOR APPENDIX IX

CONSTITUENTS

Prepared by:

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CIBA-GEIGY
ORGANIC REGION I WORKSHEET

Background: The term hazardous constituent used in the Solid Waste Disposal Act Section 3004(u) means constituents found in Appendix VIII to 40CFR part 261. EPA also defines those constituents identified in Appendix IX to 40CFR part 264. Appendix IX constituents generally constitutes a subset of Appendix VIII particularly suitable for ground water analyses. ~~However, it also includes additional constituents not found in Appendix VIII, but commonly addressed in ground water analyses conducted as part of Superfund cleanups.~~ ✓

WPT
drop here

In general, where very little is known of waste characteristics, and where there is a potential for a wide spectrum of wastes to have been released, only then is the owner/operator required to perform an extensive routine analysis for a broader spectrum of waste such as an Appendix IX analysis.

Radian Corporation of Austin, Texas, has been sub-contracted by WCC to analyze the 232 hazardous constituents in Appendix IX and will be utilizing the following SW-846 procedures listed in Table I. As such, the enclosed Organic Region I Data Validation Worksheets have been modified accordingly for each fraction to conform to the QA/QC criteria of each SW-846 test methods in Table I.

Where appropriate, action levels based on promulgated standards (e.g., Maximum Contaminant Levels (MCLs)) established under the Safe Drinking Water Act have been taken into consideration. Specifically, for Volatile Organic analyses of the ground water matrix, the detection limits for SW-846 Method 8240 are below the federal and state (Rhode Island) listed MCLs.

TABLE I
SELECTED ANALYTICAL METHODS FOR
ORGANIC APPENDIX IX ANALYSES

SW-846 Method	General Category/ Analyte	Technique	Number of Analytes Measured
6010	Metals	IGP	11
7041	Antimony	GEAA	1
7060	Arsenic	GFAA	1
7421	Lead	GFAA	1
7470	Mercury	CVAA	1
7740	Selenium	GFAA	1
7841	Thallium	GEAA	1
8080	Organochlorine Pesticides and PCBs	GC/ECD	28
8140	Organophosphorus Pesticides	GC/FPD	9
8150	Herbicides	GC/ECD	4
8240	Volatile Organics	GC/MS	* 54
8270	Semivolatile Organics	GC/MS	** 111
8280	Dioxins and Furans	GC/MS	712
9012	Cyanide	Colorimetric	1
9030	Sulfide	Titrimetric	1
TOTAL			206

~~IGP - Inductively Coupled Plasma Spectrometry~~
~~GEAA - Graphite Furnace Atomic Absorption Spectrometry~~
 GC/ECD - Gas Chromatography/Electron Capture Detection
 GC/EPD - Gas Chromatography/Flame Photometric Detection
 GC/MS - Gas Chromatography/Mass Spectrometry

- * This number includes three analytes (1,4-Dioxane, isobutanol, methacrylonitrile) that will be analyzed by Method 8240 Direct Injection.
 ** This number includes Appendix IX analytes, however, this number will increase based on site specific compounds that will be analyzed by Method 8270, also. Sym-Trinitrobenzene will be analyzed as a tentatively identified compound due to unavailability of standard.

DATA SUMMARY FORM: VOLATILES 1

Appendix IX
WATER SAMPLES

(µg/L)

Site Name: _____

Sample #: _____ Sampling Date(s): _____

Reference: _____

FRANK - completed
VOA summary form
currently generating for other
locations - JPLTo calculate sample quantitation limit:
(CRL * Dilution Factor)
PQL

PQL CRL	COMPOUND	Sample No. Dilution Factor Location																	
		Conc.	Q	Conc.	Q	Conc.	Q	Conc.	Q	Conc.	Q	Conc.	Q	Conc.	Q	Conc.	Q	Conc.	Q
10	Chloromethane																		
10	Bromomethane																		
10	*Vinyl Chloride																		
10	Chloroethane																		
10	*Methylene Chloride																		
100	Acetone																		
5	Carbon Disulfide																		
5	*1,1-Dichloroethene																		
5	1,1-Dichloroethane																		
5	*Total 1,2-Dichloroethene																		
5	Chloroform																		
5	*1,2-Dichloroethane																		
100	*2-Butanone (NEK)																		
5	*1,1,1-Trichloroethane																		
5	*Carbon Tetrachloride																		
10	Vinyl Acetate																		
5	Bromodichloromethane																		
75	ACROLEIN																		
50	ACRYLONITRILE																		
20	ACETONITRILE																		
5	3-CHLOROPROPENE																		
25	2-CHLORO-1,3-BUTADIENE																		
0.000	1,4-DIOXANE																		
20	DICHLORODIFLUOROMETHANE																		

QL = Contract Required Quantitation Limit

PQL = Practical Quantitation Limit

Q = data validation quality

Action Level Exists

SEE NARRATIVE FOR CODE DEFINITIONS

revised 07/90

10

Prepared by: JPL (Woodward-Clyde)

DATA SUMMARY FORM: VOLATILES 2

Appendix IX
 WATER SAMPLES
 (µg/L)

Site Name: _____

Case #: _____ Sampling Date(s): _____

2 times

To calculate sample quantitation limit:
 $(CRL \times \text{Dilution Factor})$
 PQL

Sample No. Dilution Factor Location															
AQL CRL	COMPOUND	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q
5	*1,2-Dichloropropane														
5	Cis-1,3-Dichloropropane														
5	Trichloroethene														
5	Dibromochloromethane														
5	1,1,2-Trichloroethane														
5	*Benzene														
5	Trans-1,3-Dichloropropene														
5	Bromoform														
50/8	4-Methyl-2-pentanone														
50/8	2-Hexanone														
5	*Tetrachloroethene														
5	1,1,2,2-Tetrachloroethane														
5	*Toluene														
5	*Chlorobenzene														
5	*Ethylbenzene														
5	*Styrene														
5	*Total Xylenes														
5	TRANS-1,2-DICHLOROETHENE														
10	1,2-DIBROMOETHANE														
10	TRANS-1,4-DICHLORO-2-BUTENE														
20	1,2-DIBROMO-3-CHLOROPROPANE														
10	ETHYL METHACRYLATE														
5	ISOMETHANE														
10,000	ISOBUTANOL														

CRL = Contract Required Quantitation Limit

*Action Level Exists

SEE NARRATIVE FOR CODE DEFINITION

revised 07/9
10

PQL = Practical Quantitation Limit

Q = Data Validation Qualifier

Prepared by: JPL (Woodward-Clyde)

DATA SUMMARY FORM: VOLATILE ORGANICS 3

Appendix IX
WATER SAMPLES
(µg/L)

Site Name: _____

Case #: _____ Sampling Date(s): _____

Reference

To calculate sample quantitation limit
(QL * Dilution Factor)

[illegible]

PQL = Quantitation Limit
PRACTICAL

Q = data validation quality

SEE NARRATIVE FOR CODE DEFINITION
revised 07/90

DATA SUMMARY FORM: VOLATILES 1

Appendix II
SOIL SAMPLES
(µg/Kg)

to Name: _____

to #1: _____ Sampling Date(s): _____

terrace

To calculate sample quantitation limit:
(~~CRL~~ * Dilution Factor) / ((100 - % moisture)/100)
PQL

PQL CRL	COMPOUND	Sample No.		Dilution Factor		X Moisture		Location											
		conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q
2018	Chloromethane																		
2018	Bromomethane																		
2018	Vinyl Chloride																		
2018	Chloroethane																		
2018	Methylene Chloride																		
2018	Acetone																		
2018	Carbon Disulfide																		
2018	1,1-Dichloroethene																		
2018	1,1-Dichloroethane																		
2018	1,2-Dichloroethene																		
2018	Chloroform																		
2018	1,2-Dichloroethane																		
2018	2-Butanone (MEK)																		
2018	1,1,1-Trichloroethane																		
2018	Carbon Tetrachloride																		
2018	Vinyl Acetate																		
2018	Bromodichloromethane																		
1500	ACROLEIN																		
1000	ACRYLONITRILE																		
400	ACETONITRILE																		
100	3-CHLOROPROPENE																		
500	2-CHLORO-1,3-BUTADIENE																		
1000	1,4-DIOXANE																		
400	DICHLOROFUOROMETHANE																		

PQL = Contract Required Quantitation Limit

PQL = Practical Quantitation Limit (wet-weight basis): samples results on
a dry weight basis will yield higher PQL (refer to calculation: top right)

SEE NARRATIVE FOR CODE DEFINITIONS

Q = data validation qualifier

revised 07/90
10

Prepared by: JPL/Woodward-Clay

DATA SUMMARY FORM: VOLATILES 2

Appendix II

SOIL SAMPLES

(µg/Kg)

Site Name: _____

Case #: _____ Sampling Date(s): _____

Reference

To calculate sample quantitation limit:
 $(CRL \cdot \text{Dilution Factor}) / ((100 - \% \text{ moisture})/100)$
PQL

PQL CRQL	COMPOUND	Sample No.		Dilution Factor		% Moisture		Location													
		conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q	conc.	Q
100.5	1,2-Dichloropropane																				
100.5	Cis-1,3-Dichloropropene																				
100.5	Trichloroethene																				
100.5	Dibromochloromethane																				
100.5	1,1,2-Trichloroethane																				
100.5	Benzene																				
100.5	Trans-1,3-Dichloropropene																				
100.5	Bromoform (TRIBROMOMETHANE)																				
100.5	4-Methyl-2-pentanone																				
100.5	2-Hexanone																				
100.5	Tetrachloroethene																				
100.5	1,1,2,2-Tetrachloroethane																				
100.5	Toluene																				
100.5	Chlorobenzene																				
100.5	Ethylbenzene																				
100.5	Styrene																				
100.5	Total Xylenes																				
100	TRANS-1,2-DICHLOROETHENE																				
200	1,2-DIBROMOETHANE																				
200	TRANS-1,4-DICHLORO-2-BUTENE																				
100	1,2-DIBROMO-3-CHLOROPROPANE																				
200	ETHYL METHACRYLATE																				
100	ISOMETHANE																				
10,000	ISOBUTANOL																				

CRQL = Contract Required Quantitation Limit

SEE NARRATIVE FOR CODE DEFINITIONS

PQL = Practical Quantitation Limit (wet-weight basis): sample results on a dry weight basis will yield a higher PQL (refer to calibration: top right)

Q = Data Validation Qualifier revised DT/9C 10

DATA SUMMARY FORM: *VOLATILE* ORGANICS 3

Appendix IX
SOIL SAMPLES
(µg/Kg)

Item Name: _____

~~Site #:~~ _____ Sampling Date(s): _____

Reference

To calculate sample quantitation limit
(QL = Dilution Factor) / ((100 - % moisture)/100)

[illegible]

→ QL = Quantitation Limit
PRACTICAL

Q = data validasi kualitatif

SEE NARRATIVE FOR CODE DEFINITION
revised DT/5
18

CIBA-GEIGY
REGION I
Data Review Worksheets
Re-Edited for Appendix IX
Constituent Analysis

Site Name
Reference No

REGION I REVIEW OF ORGANIC
CONTRACT LABORATORY DATA PACKAGE

The hardcopied (laboratory name) _____ data package received at Region I has been reviewed and the quality assurance and performance data summarized. The data review included:

~~Case No.:~~

Matrix:

~~SDC No.:~~

No. of Samples:

Sampling Date(s):
Shipping Date(s):
Date Rec'd by Lab:

~~Sample Identification:~~
~~Traffic Report Nos.:~~

Trip Blank No.:
Equipment Blank No.:
Field Dup Nos.:

Add after
field dup nos.

SW-846 (3rd Edition)

~~SW-846 (3rd Edition) requires that specific analytical work be done and that associated reports be provided by the laboratory to the Regions, EMSL, LV, and SMO. The general criteria used to determine the performance were based on an examination of:~~

- | | |
|-----------------------|--------------------------------|
| -Data Completeness | -Matrix Spike/Matrix Spike Dup |
| -Holding Times | -Field Duplicates |
| -GC/MS Tuning | -Internal Standard Performance |
| -Calibrations | -Pesticide Inst. Performance |
| -Blanks | -Compound Identification |
| -Surrogate Recoveries | -Compound Quantification |

Overall Comments:

Definitions and Qualifiers:

- A - Acceptable data.
J - Approximate data due to quality control criteria.
R - Reject data due to quality control criteria
U - Analyte not detected

Reviewer:

Date:

REGION I
Data Review Worksheets

I. DATA COMPLETENESS

MISSING INFORMATION

DATE LAB CONTACTED

DATE REC'D

REGION I
Data Review Worksheets

II. HOLDING TIMES

Complete table for all samples and circle the fractions which are not within criteria.

SAMPLE ID	DATE SAMPLED	VOA	BNA	DATE EXTR	DATE ANAL	PEST	DATE ANAL
		DATE ANAL				DATE EXTR	

In accordance with Table 4-1 of Section Four of SW-846 and EPA Region I protocols

VOA - Unpreserved: Aromatic within 7 days, non-aromatic within 14 days of sample collection.

Preserved: Both within 14 days of sample collection.

Soils: Both within 14 days of sample collection.

Water: in accordance with Method 8270

BNA & PEST - *Extracted within 7 days, analyzed within 40 days, soils and water.*

Soils (Solids): Extracted within 14 days, analyzed within 40 days.

ACTION: 1. If holding times are exceeded all positive results are estimate (J) and non-detects are estimated (UJ). 2. If holding times are grossly exceeded, the reviewer may determine that non-detects are unusable(*).

in accordance with Method 8270

REGION I
Data Review Worksheets

III. GC/MS TUNING

The DFTPP performance results were reviewed and found to be with the specified criteria.

If no,
Samples affected:

The BFB performance results were reviewed and found to be within the specified criteria.

If no,
Samples affected:

If mass calibration is in error refer to the Region guidelines for expanded criteria. If necessary, all associated data as unusable (R).

*Note: BFB and DFTPP tune criteria for CLP and SW 846
are the same @*

REGION I
Data Review Worksheet

IV A. ~~SEMIVOLATILE~~ CALIBRATION VERIFICATION
VOLATILE

Date of Initial Calibration :
Dates of Continuing Calibrations :
Instrument ID :
Matrix/Level :

<u>DATE</u>	<u>CRITERIA OUT</u> <u>RF, %RSD, RF, %D</u>	<u>COMPOUND (VALUE)</u>
-------------	--	-------------------------

Samples Affected:

Samples Affected:

Samples Affected:

Samples Affected:

Samples Affected:

Samples Affected:

Samples Affected:

Samples Affected:

Samples Affected:

Samples Affected:

1. All RF's ~~and RF's~~ must be >0.05
2. All %RSD's must be $<30\%$
3. All %D's must be $<25\%$

ACTION:

1. If any compound has an initial RF or a continuing RF of <0.05 :
 - a. Flag positive results for that compound as estimated (J).
 - b. Flag non-detects for that compound as unusable (R).
 2. If any compound has a %RSD $>30\%$ or a %D $>25\%$:
 - a. Flag positive results for that compound as estimate (J).
 - b. Flag non-detects for that compound as estimated (UJ) if the %RSD or %D is $>50\%$.
- for volatiles and $>30\%$
for semi-volatiles*

A separate worksheet should be filled out for each initial curve.

REGION I
Data Review Worksheet

IV B. SEMIVOLATILE CALIBRATION VERIFICATION

Date of Initial Calibration :
Dates of Continuing Calibrations :
Instrument ID :

<u>DATE</u>	<u>CRITERIA OUT</u> <u>RF,%RSD,RF,%D</u>	<u>COMPOUND</u>
-------------	---	-----------------

____ Samples Affected:

____ Samples Affected:

____ Samples Affected:

____ Samples Affected:

____ Samples Affected:

____ Samples Affected:

____ Samples Affected:

____ Samples Affected:

____ Samples Affected:

____ Samples Affected:

____ Samples Affected:

____ Samples Affected:

____ Samples Affected:

____ Samples Affected:

See worksheet IV-A for criteria and actions.

A new worksheet should be filled out for each initial curve.

REGION I
Data Review Worksheet

V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

List the contamination in the blanks below.

1. Laboratory Blanks

LEVEL: _____

<u>DATE</u>	<u>LAB ID</u>	<u>FRACTION/ MATRIX</u>	<u>COMPOUND</u>	<u>CONCENTRATION/ UNITS</u>
-------------	---------------	-----------------------------	-----------------	---------------------------------

2. Equipment (Field) and Trip Blanks

<u>DATE</u>	<u>LAB ID</u>	<u>FRACTION/ MATRIX</u>	<u>COMPOUND</u>	<u>CONCENTRATION/ UNITS</u>
-------------	---------------	-----------------------------	-----------------	---------------------------------

A separate worksheet should be used for low and medium level blanks.

V B. BLANK ANALYSIS RESULTS (Section 3)

3. Blank actions

Action levels should be based upon the highest concentration of contaminant determined in any blank. The action level for samples which have been concentrated or diluted should be multiplied by the concentration/dilution factor. No positive sample result should be reported unless the concentration of the compound in the sample exceeds the action level of 10 x's the amount for any other compound. Specific actions are as follows:

1. The concentration is less than the CRQL, report the CRQL.
2. The concentration is greater than the CRQL, but less than the action level, report the concentration found U.
3. The concentration is greater than the action level, report the concentration unqualified.

For examples refer to the Regional Guidelines.

Common contaminants = methylene chloride, acetone, 2-butanone, toluene, and base-neutral fraction phthalate ester compounds (i.e., bis(2-ethyl hexyl)phthalate.

LEVEL: _____

<u>COMPOUND</u>	<u>MAX. CONC./</u> <u>UNITS</u>	<u>ACTION LEVEL/</u> <u>UNITS</u>	<u>CRQL</u>
-----------------	------------------------------------	--------------------------------------	-------------

A separate worksheet should be used for low and medium level blanks.

VI. SURROGATE SPIKE RECOVERIES

List the percent recoveries which do not meet the criteria for surrogate recovery.

Matrix: _____

TR #'S	TOL	VOA BFB	DC ^E	NBZ	B/N FBP	TPH	PHL	A 2FP	TBP	PEST DBC*
QC Limits	88	86	76	35	43	33	30	21	10	24
(WATERS)	to 110	to 115	to 114	to 114	to 116	to 141	to 94	to 100	to 123	to 154
(SOLIDS)	84	59	70	23	30	18	24	25	19	20
Surrogate Actions:	138	113	121	120	115	137	113	121	122	150

* - Advisory only

* - Advisory only

PERCENT RECOVERY

<10%	10% - CRR	R(NIN)	R(MAX)
J	J	J	J
R	UJ	A	A

Positive sample results

Non-detected results

R(NIN): Denotes lower limit of surrogate recovery range window (i.e., TOL

CRR = Contract Required Recovery Range.

Surrogate action should be applied:

1. If at least two surrogates in a B/N or A fraction or one surrogate in the VOA fraction are out of specification, but have recoveries of >10%.
2. If any one surrogate in a fraction shows <10% recovery.

which denotes the surrogate standard tolerance (±8%) has R(NIN) of 88% for a water matrix sample and a R(MAX) of 110. The acceptance window is therefore 88-110%.

VII A. MATRIX SPIKE/MATRIX SPIKE DUPLICATE

1. Matrix Spike/Matrix Spike Duplicate Recoveries and Precision

TR Nos. _____, _____ Level: _____ Matrix: _____

List the percent recoveries and RPD's of compounds which do not meet the criteria stated ~~on Form 3~~ *in SW-846* ~~on Form 3~~ *recovery and RPD maximums on a EPA Form III.*
supply the new SW-846 percent

<u>FRACTION/ MS OR MSD</u>	<u>COMPOUND</u>	<u>% REC/RPD</u>	<u>QC LIMITS</u>
			<i>Contract Radon Corporation (the laboratory) if the revised Form III is not present</i>

QUALIFICATION IS LIMITED TO THE UNSPIKED SAMPLE ONLY.

1. If any compound does not meet the ~~Contract Required~~ Recovery range (CRR) follow the actions stated below:

	PERCENT RECOVERY			MIN	R(MAX)
	<10%	10%-CRR	CRR		
	J	J	J		
Positive sample results					
Non-detected results					
<i>R(MIN): Denotes lower limit of recovery range window (i.e., trichloroethene: 71%)</i>					

2. If any compound does not meet the RPD criteria, flag positive MIN results for that compound as estimated (J).

A separate worksheet should be used for each MS/MSD pair.

Refer to Appendix A for Matrix spike ~~to~~ *percent* RPD maximums and percent recoveries.

*R(MAX)
for
trichloroethene
(water mix)
is 120%
The acceptance
window is
therefore 71-120%.*

VII B. MATRIX SPIKE/MATRIX SPIKE DUPLICATE (Section 2)

3. Matrix Spike Duplicate - Unspiked Compounds

TR Nos. _____, _____

List the concentrations of the unspiked compounds and determine the percent RSD's of the unspiked sample, matrix spike, and matrix spike duplicate. No limits have been developed for the RSD values of the unspiked compounds.

<u>FRACTION</u>	<u>COMPOUND</u>	<u>SAMPLE, MS, MSD CONC.</u>	<u>%RSD</u>
-----------------	-----------------	------------------------------	-------------

The reviewer must use professional judgment to determine if there is a need to qualify any of the unspiked compounds in the sample.

VIII. FIELD DUPLICATE PRECISION

TR Nos. _____, _____ Matrix: _____

List the concentrations of the compounds which do not meet the following RPD criteria:

1. An RPD of <30% for water duplicates.
2. An RPD of <50% for soil duplicates.

<u>FRACTION</u>	<u>COMPOUND</u>	<u>SAMPLE CONCDUP</u>	<u>SAMPLE CONC</u>	<u>RPD</u> ✓
-----------------	-----------------	-----------------------	--------------------	--------------

ACTIONS:

1. If the results for any compounds do not meet the RPD criteria, flag the positive results for that compound as estimated.
2. If one value is non-detected, and one is above the CRQL:
 - a. Flag the positive result as estimated (J).
 - b. Flag the non-detected result as estimated (UJ).

NOTE: Professional judgment may be utilized to apply duplicate action to all samples of a similar matrix.

A separate worksheet should be filled out for each field duplicate pairs.

IX. INTERNAL STANDARD PERFORMANCE

List the internal standard areas of samples which do not meet the criteria of +100% or -50% of the internal standard area in the associated continuing calibration standard.

<u>SAMPLE ID</u>	<u>DATE</u>	<u>IS OUT</u>	<u>IS AREA/ RT</u>	<u>ACCEPTABLE RANGE</u>	<u>ACTION</u>
------------------	-------------	---------------	------------------------	-------------------------	---------------

ACTION:

1. If an IS area count is outside the criteria -50% or +100% of the associated standard:
 - a. Positive results for compounds quantitated using that IS are flagged as estimated (J) for that sample fraction.
 - b. Non-detects for compounds quantitated using that IS are flagged as estimated (UJ) for that sample fraction.
 - c. If extremely low area counts are reported, or if performance exhibits a major drop-off, then a severe loss of sensitivity is indicated. Non-detects should then be flagged as unusable (R).
2. If an IS retention time varies more than 30 seconds, the chromatographic profile for that sample must be examined to determine if any false positives or negatives exist. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for that sample fraction.

X A. PESTICIDE INSTRUMENT PERFORMANCE (Section 1)

1. DDT Retention Time

List the DDT standards which have a retention time (RT) of less than 12 minutes on the packed column (except OV-1 or OV-101).

Note: A megabore or capillary column standard RT may be shorter than 12 minutes.

<u>STANDARD ID</u>	<u>DATE/ TIME</u>	<u>RT</u>	<u>SAMPLES AFFECTED</u>	<u>ACTIONS</u>
--------------------	-----------------------	-----------	-------------------------	----------------

ACTION:

If the RT is less than 12 minutes, examine the chromatography to evaluate the separation. If adequate separation is not achieved, flag all affected compound data as unusable (R).

X B. PESTICIDE INSTRUMENT PERFORMANCE (Section 2)

2. Retention Time Windows

List the compounds which are not within the established windows.

<u>COMPOUND</u>	<u>DATE</u> <u>(TIME)</u>	<u>RT/RT WINDOW</u>	<u>SAMPLES AFFECTED</u>
		1 WP - spot apart	

Check the sample chromatograms of the samples analyzed after the last in control standard for peaks within an expanded window. If no peaks are present, there is usually no effect on the data. Refer to Regional guidelines for information on qualifying data if peaks are present. If peaks are present, discuss actions below:

REGION I
Data Review Worksheets

X C. PESTICIDE INSTRUMENT PERFORMANCE (Section 3)

3. DDT and Endrin Degradation

List the standards which have a DDT or Endrin breakdown of greater than 20%.

<u>STANDARD ID</u>	<u>DDT OR ENDRIN</u>	<u>PERCENT BREAKDOWN</u>	<u>ENDRIN SAMPLES AFFECTED</u>	<u>DDD, DDE OR ENDRIN KETONE PRESENT</u>
------------------------	--------------------------	------------------------------	------------------------------------	--

If the percent breakdown for DDT is greater than 20%.

1. Flag all positive results for DDT as estimated (J) for all samples following the last in control standard. If no DDT was present, but DDD and/or DDE are positive, then flag the quantification limit for DDT as unusable (R).
2. Flag all positive results for DDD +/-or DDE as estimated (J).

If the percent breakdown for Endrin is greater than 20%:

1. Flag all positive results for endrin as estimated (J) for all samples following the last in-control standard. If no endrin was detected, but endrin aldehyde and/or endrin ketone are positive, flag the quantification limit for endrin as unusable (R).
2. Flag all positive results for endrin ketone as estimated (J).

X D. PESTICIDE INSTRUMENT PERFORMANCE (Section 4)

4. DBC Retention Time Check

List the percent difference for the DBC shift greater than 2% for packed columns, greater than 1.5% for wide-bore capillary columns, or greater than 0.3% for narrow-bore capillary columns.

<u>TR #'s</u>	<u>DBC % DIFFERENCE</u>	<u>ACTIONS</u>
---------------	-------------------------	----------------

If the DBC does not meet the retention time criteria, the analysis may be flagged as unusable (R) for the affected samples, but qualification of the data is left up to the professional judgment of the reviewer. Discuss any qualification of the data below:

XI A. PESTICIDE CALIBRATION (Sections 1 and 2)

1. Initial Calibration

List the compounds which did not meet the Relative Standard Deviation (RSD) criteria of less than ~~10%~~ ²⁰ for the initial calibration on the quantification column.

<u>DATE</u>	<u>COMPOUND</u>	<u>%RSD</u>	<u>COLUMN</u>	<u>SAMPLES AFFECTED</u>
-------------	-----------------	-------------	---------------	-------------------------

Flag all associated positive results as estimated (J) for samples which did not meet the %RSD criteria.

2. Analytical Sequence

Did the laboratory follow the correct ~~the correct~~ ^{supply the analytical utilized and the} sequence described in appropriate ~~the SW?~~ ^{retention time windows} Yes or No

If no,

The data may be affected. The data reviewer must use professional ~~for~~ judgment to determine the severity of the effect and qualify the data ~~and~~ accordingly. Discuss any actions below:

Refer to Method 8000 protocols for guidance on any action taken.

Contact the laboratory ^{discuss} to ~~contact~~ any and anomalies on future analyses encountered to prevent recurrence

each analyze in the check standard as per the requirements of Method 8000 in SW-846 and the Radionuclide ^{3rd} Edition

QAPP

XI B. PESTICIDE CALIBRATION (Section 3)

3. Continuing Calibration

List the compounds [±] which did not meet the percent difference (%D) criteria of ~~±15%~~ on the quantification column or ^{±20%} ~~±20%~~ on the confirmation for the continuing calibration.

<u>DATE</u>	<u>COMPOUND</u>	<u>%D</u>	<u>COLUMN</u>	<u>SAMPLES AFFECTED</u>
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

If the %D criteria is not met, flag all associated positive results as estimated (J).

XII. SAMPLE QUANTIFICATION

In the space below, please show a minimum of one sample calculation per fraction:

VOA:

BNA:

PEST/PCB:

Additional
 Appendix II Method specific validation checks involving
 Method 8080 (Organochlorine Pesticide and PCBs), Method
 8140 (Organophosphorus Pesticides) and Method
 8150 (Herbicides).

A. Method 8150's surrogate standard is 2,4-dichlorophenyl
 acetic acid and 8140's is triphenylphosphate

List the percent recovery: %
 List the QC limits listed on the Summary Form: %
 Surrogate Actions:

	Percent Recovery		R(MAX) QCR
	< 10%	10% - QCR R(MIN)	
Positive Sample Results	J	J	J
Non-detected Results	R	UJ	A

Note: The laboratory must report an acceptable surrogate recovery range as per the
 requirements of Section 8.3 of Methods 8140 and 8150. As such, the Method 8150
 surrogate spike acceptance window is 50-150% for both waters and solids. The R(MIN) is
 50% and R(MAX) is 150%. Method 8140's surrogate is triphenylphosphate. For waters
 the acceptance window is 40-140% (R(MIN): 40; R(MAX): 140) and for solids the acceptance
 window is 42-154% (R(MIN): 42; R(MAX): 154)@

B. Method 8140's MS/MSD compounds are: dieldrin,
 ethyl parathion, famphur, methyl parathion and
 phorate. As an example,
 Method 8150's MS/MSD compounds are
 2,4-D, ²lincolb, 2,4,5-T and 2,4,5-TP (Silvex).
 List the percent recoveries and RPD's of compounds
 which do not meet the criteria listed on EPA Form III
 by Radian (laboratory).

Fraction	% Rec	RPD	
MS or MSD	Compound	% REC	QC Limits

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QUALIFICATION IS LIMITED TO THE UNSPIRED SAMPLE ONLY.

1. If any compound does not meet the Contract Required Recovery range (CRR) follow the actions stated below:

	PERCENT RECOVERY		
	$<101 \frac{101 - CRR(NM)}{J}$	$101 \frac{101 - CRR(NM)}{J}$	$\frac{R(NM)}{A}$
Positive sample results	J	J	J
Non-detected results	R	UJ	A

2. If any compound does not meet the RPD criteria, flag positive results for that compound as estimated (J).

A separate worksheet should be used for each MS/MSD pair.

Standard below are the Matrix spike (R10 maximums and accuracy) and Surrogate Recovery:

Parameter	Matrix Spike(a)				Surrogate Spike Accuracy(c)	
	Precision RPD % (b)		Accuracy % of Recovery		% of Recovery	
	Water	Solids	Water	Solids	Water	Solids
SW846, Method 8150 Herbicides: (3rd)						
2,4-D	N/A 13	N/A	69-159	N/A	-	-
Dinoseb	N/A 15	N/A	N/A 58-150	N/A	-	-
2,4,5-TP	N/A 25	N/A	53-103 29-82	N/A	-	-
2,4,5-T	N/A 9	N/A	60-110 82-138	N/A	-	-
2,4-Dichlorophenylacetic acid	-	-	-	-	50-150	50-150

- (a) Matrix spike precision and accuracy goals, where stated, are found in EPA method references and will be used as starting points. Limits developed in-house will be used and updated throughout the program.
- (b) "RPD" = relative percent difference. Precision is expressed according to the type of measurement (i.e., field duplicates precision is expressed as the RPD between duplicate results).
- (c) Accuracy goals stated are from EPA CLP, SW-846 methods, or Radian-derived limits. These limits will be used as starting points for control chart generation. Limits developed in-house will be used and updated throughout the program.

N/A Not available (To be developed by Radian)

Parameter	Standard Deviation		Accuracy (a) % of Recovery		Surrogate Spike Accuracy (a) % of Recovery	
	Water	Solids	Water	Solids	Water	Solids
<i>SW 816 Marked 81404 Organophosphorus Pesticides</i>						
Organophosphorus Pesticides:						
Dimethoate	N/A	N/A	N/A	N/A	-	-
Famphur	N/A 50	N/A 33	10-168	0-180 10-136	-	-
Ethylparathion	N/A 14	N/A 21	20-152 15-112	0-188 29-133	-	-
Thionazin	N/A	N/A	N/A	N/A	-	-
Disulfoton	9.0 36	N/A 39	10-138	0-164	-	-
Methyl Parathion	5.3 35	N/A 36	¹⁰⁻²¹³ 16-158	3-147 10-219	-	-
Phorate	8.9 32	N/A 33	¹⁰⁻¹⁸³ 14-116	3-165 10-177	-	-
Sulfotepp	N/A	N/A	32-124	7-165 10-166	-	-
Triphenylphosphate	-	-	-	-	40-140 23-176	42-154 28-174

(a) These limits were developed in-house. Radian will update these limits throughout the program.

N/A Not available (*To be developed by Radian*)

XIV. Dioxin (Method 8280: Full Scan Taria - Oct+A including 2,3,7,8-TCDD)

Refer to Appendix B for Radcan Dioxin Summary Forms. QA/QC criteria for dioxin are listed below.

(Ciba being inserted)

Other
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PCDD AND PCDF (DIOXINS AND FURANS)
QUALITY CONTROL OBJECTIVES

Parameter	Precision RPD % for Duplicate Analyses(a) Water	Completeness Expected Completeness %
PCDD	60-140	90
PCDF	60-140	90

(a) These objectives are for recovery check samples, it is anticipated that the field samples will fall within these objectives.

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Page 21 of 24

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Reference

ORGANIC REGIONAL DATA ASSESSMENT

CASE NO.:

LABORATORY:

SDG #:

SW 846 ~~SON, #~~ 3rd Edition

~~DPO: ACTION:~~

~~FVI:~~

SITE: CIBA-GEIGY, CRANSTON, RI

NO. OF SAMPLES/ MATRIX:

REVIEWER (IF NOT ESD):

REVIEWER'S NAME:

COMPLETION DATE:

DATA ASSESSMENT SUMMARY

	<u>VOA</u>	<u>BNA</u>	<u>PEST</u>	<u>OTHER</u>
1. HOLDING TIMES				
2. GC/MS TUNE/INSTR. PERFORMANCE				
3. CALIBRATIONS				
4. BLANKS				
5. SURROGATES				
6. MATRIX SPIKE/DUP				
7. OTHER QC				
8. INTERNAL STANDARDS				
9. COMPOUND IDENTIFICATION				
10. SYSTEM PERFORMANCE				
11. OVERALL ASSESSMENT				

O = Data had no problems/or qualified due to minor problems.

M = Data qualified due to major problems.

Z = Data unacceptable.

X = Problems, but do not affect data.

ACTIONS

ITEMS:

AREAS OF
CONCERN:

NOTABLE
PERFORMANCE:

Appendix A

Matrix Spike RPD Maximums

and Percent Recoveries

Parameter	Matrix Spike(a)			
	Precision		Accuracy	
	RPD % (b)		% of Recovery	
	Water	Solids	Water	Solids
Volatiles:				
Trichloroethene	14	24	71-120	62-137
Benzene	11	21	76-127	66-142
Toluene	13	21	76-125	59-139
Chlorobenzene	13	21	75-130	60-133
1,1-Dichloroethene	14	22	61-145	59-172
Toluene-d8	-	-	-	-
4-Bromofluorobenzene	-	-	-	-
1,2-Dichloroethane-d4	-	-	-	-

- (a) Matrix spike precision and accuracy goals, where stated, are found in EPA method references and will be used as starting points. Limits developed in-house will be used and updated throughout the program.
- (b) "RPD" = relative percent difference. Precision is expressed according to the type of measurement (i.e., for field duplicates precision is expressed as the RPD between duplicate results).

Parameter	Matrix Spike(a) (d)			
	Precision		Accuracy	
	RPD % (b)		% of Recovery	
	Water	Solids	Water	Solids
<u>GC/MS Semivolatiles:</u>				
Phenol	42	35	12-89	26-90
2-Chlorophenol	40	50	27-123	25-102
1,4-Dichlorobenzene	28	27	36-97	28-104
N-Nitroso-di-n-propyl-amine	38	38	41-116	41-126
1,2,4-Trichlorobenzene	28	23	39-98	38-107
4-Chloro-3-methylphenol	42	33	23-97	26-103
Acenaphthylene	31	39	46-118	31-137
4-Nitrophenol	50	50	10-80	11-114
2,4-Dinitrotoluene	38	47	24-96	28-89
Pentachlorophenol	50	47	9-103	17-109
Pyrene	31	36	26-127	35-142
Nitrobenzene-d5	-	-	-	-
2-Fluorobiphenyl	-	-	-	-
p-Terphenyl-d14	-	-	-	-
Phenol-d5	-	-	-	-
2-Fluorophenol	-	-	-	-
2,4,6-Tribromophenol	-	-	-	-
Propazine	N/A 18*	N/A 5*	61-85 34-117	68-94 77-100*
Tinovin-327	N/A 15*	N/A 6*	81-143	112-155 77-107*
			47-123	

- (a) Matrix spike precision and accuracy goals, where stated, are found in EPA method references and will be used as starting points. Limits developed in-house will be used and updated throughout the program.
- (b) "RPD" = relative percent difference. Precision is expressed according to the type of measurement (i.e., for field duplicates precision is expressed as the RPD between duplicate results).
- (c) Accuracy goals stated are from EPA CAP, SW-846 methods, or Radian-derived limits. These limits will be used as starting points for control chart generation. Limits developed in-house will be used and updated throughout the program.
- (d) Accuracy goals stated for Propazine and Tinovin-327 were calculated using the data obtained from the Precision and Accuracy study originally performed by ATAS for the fingerprint compounds of interest. Since Radian has no experience with these compounds, these ranges may vary slightly.
- * These values were calculated based on data collected from the reagent spike blanks and fine sediment samples. These data will be updated as more data points become available throughout Round I.
- N/A Not available.

Parameter	Matrix Spike(a)			
	Precision		Accuracy	
	RPD % (b)		% of Recovery	
	Water	Solids	Water	Solids
Pesticides/Polychlorinated				
Biphenyls:				
gamma-BHC	15	50	56-123	46-127
Heptachlor	20	31	40-131	35-130
Aldrin	22	43	40-120	34-132
Dieldrin	18	38	52-126	31-134
Endrin	21	45	56-121	42-139
4,4'-DDT	27	50	38-127	23-134
Dibutylchlorodane	-	-	-	-

- (a) Matrix spike precision and accuracy goals, where stated, are found in EPA method references and will be used as starting points. Limits developed in-house will be used and updated throughout the program.
- (b) "RPD" = relative percent difference. Precision is expressed according to the type of measurement (i.e., for field duplicates precision is expressed as the RPD between duplicate results).
- (c) Accuracy goals stated are from EPA CLP, SW-846 methods, or Radion-derived limits. These limits will be used as starting points for control chart generation. Limits developed in-house will be used and updated throughout the program.

Appendix B

Dioxin Summary Forms

Method 8280 (Full-Scan)

Tetra - OCTA

(Qualification actions for dioxins
for each table enclosed to
be incorporated by WCC - Ciba Geigy)

INTERNAL STANDARD RECOVERY

[illegible]

*C-TCDD : Carbon 13 labeled 2,3,7,8-tetrachlorodibenzodioxin

*C-HxCDD: Carbon 13 labeled 1,2,3,6,7,8-hexachlorodibenzodioxin

*C-OCDD : Carbon 13 labeled octachlorodibenzodioxin

*C-TCDF : Carbon 13 labeled 2,3,7,8-tetrachlorodibenzofuran

*C-13pCDF: Carbon 13 labeled 1,2,3,4,6,7,8-heptachlorodibenzofuran

FORM 3
INITIAL CALIBRATION SUMMARY

INSTRUMENT								
CONC.								
TIME								
STD. ID.								
COMPOUND						RF	SD	RSD
2378 TCDD								
12378 PeCDD								
123478 HxCDD								
123678 HxCDD								
123789 HxCDD								
1234678 HpCDD								
OCDD								
2378 TCDF								
12378 PeCDF								
23478 PeCDF								
123478 HxCDF								
123678 HxCDF								
123789 HxCDF								
234678 HxCDF								
1234678 HpCDF								
1234789 HpCDF								
OCDF								
*C-TCDD ¹								
*C-TCDF ¹								
*C-HxCDD ²								
*C-HpCDF ²								
*C-OCDD ²								

IS

STANDARDS

conc. pg/ul

*C-TCDD	*13C12-2,3,7,8-TCDD	
*C-TCDF	*13C12-2,3,7,8-TCDF	
*C-HxCDD	*13C12-1,2,3,6,7,8-HxCDD	
*C-HpCDF	*13C12-1,2,3,4,6,7,8-HpCDD	
*C-OCDD	*13C12-OCDDP	

1. Based on recovery standard 13C-1234-TCDD
2. Based on recovery standard 12C-123789-HxCDD

FORM 4
CONTINUING CALIBRATION SUMMARY

INSTRUMENT

		INITIAL CURVE (RF)	CONT. CALIB. (RF)	RPD	CONT. CALIB. (RF)	RPD	CONT. CALIB. (RF)	RPD
CONC.	DATE/TIME STD.ID.							
2378	TCDD							
12378	PeCDD							
123478	HxCDD							
123678	HxCDD							
123789	HxCDD							
1234678	HpCDD							
	OCDD							
2378	TCDF							
12378	PeCDF							
23478	PeCDF							
123478	HxCDF							
123678	HxCDF							
123789	HxCDF							
234678	HxCDF							
1234678	HpCDF							
1234789	HpCDF							
	OCDF							
*C-TCDD ¹								
*C-TCDF ¹								
*C-HxCDD ²								
*C-HpCDF ²								
*C-OCDD ²								

**FORM 5
STANDARD WORKSHEET**

DATE : _____
 INJ TIME: _____
 STD ID : _____
 COLUMN : _____
 INST ID : _____

	SCAN# 'S	ION	AREA	ION	AREA*	AREA/AREA*	CONC	RRE
13C-1234-TCDD	_____	(332)	_____	(334)	_____	_____	_____	_____
13C-2378-TCDD**	_____	(332)	_____	(334)	_____	_____	_____	_____
2378-TCDD ¹	_____	(320)	_____	(322)	_____	_____	_____	_____
12378-PeCDD ¹	_____	(358)	_____	(356)	_____	_____	_____	_____
13C-123789-HxCDD	_____	(404)	_____	(402)	_____	_____	_____	_____
13C-123678-HxCDD***	_____	(404)	_____	(402)	_____	_____	_____	_____
__2378-HxCDD ²	_____	(392)	_____	(390)	_____	_____	_____	_____
1234678-HpCDD ²	_____	(426)	_____	(424)	_____	_____	_____	_____
13C-OCDD***	_____	(470)	_____	(472)	_____	_____	_____	_____
OCDD ³	_____	(458)	_____	(460)	_____	_____	_____	_____
13C-2378-TCDF**	_____	(316)	_____	(318)	_____	_____	_____	_____
2378-TCDF ⁴	_____	(304)	_____	(306)	_____	_____	_____	_____
_2378-PeCDF ⁴	_____	(342)	_____	(340)	_____	_____	_____	_____
__2378-HxCDF ⁵	_____	(376)	_____	(374)	_____	_____	_____	_____
13C-1234678-HpCDF***	_____	(422)	_____	(420)	_____	_____	_____	_____
__2378-HpCDF ⁵	_____	(410)	_____	(408)	_____	_____	_____	_____
OCDF ⁵	_____	(442)	_____	(444)	_____	_____	_____	_____

- * - Ion used for quantitation
- ** - Quantitation based on 13C-1,2,3,4-TCDD
- ***- Quantitation based on 13C-1,2,3,7,8,9-HxCDD
- 1 - Int Std 13C-2,3,7,8-TCDD
- 2 - Int Std 13C-1,2,3,6,7,8-HxCDD
- 3 - Int Std 13C-OCDD
- 4 - Int Std 13C-2,3,7,8-TCDF
- 5 - Int Std 13C-1,2,3,4,6,7,8-HpCDF

NOTE: If more than one ratio is required please write ratios directly on EICP.

FORM 6
SAMPLE WORKSHEET

DATE : _____
INJ TIME : _____
CLIENT ID : _____
SAMPLE ID : _____
SAMPLE SIZE: _____

COLUMN : _____
INST ID : _____
CURVE/STDS: _____
COMMENTS : _____

	SCAN#	ION	AREA	ION	AREA*	AREA/AREA*	CONC	RRE	DL
13C-1234-TCDD		(332)		(334)					
13C-2378-TCDD**		(332)		(334)					
2378-TCDD ¹		(320)		(322)					
TOTAL-TCDD ¹		(320)		(322)					
12378-PeCDD ¹		(358)		(356)					
TOTAL-PeCDD ¹		(358)		(356)					
13C-123789-HxCDD		(404)		(402)					
13C-123678-HxCDD***		(404)		(402)					
XX2378-HxCDD ²		(392)		(390)					
TOTAL-HxCDD ²		(392)		(390)					
1234678-HpCDD ²		(426)		(424)					
TOTAL-HpCDD ²		(426)		(424)					
13C-OCDD***		(470)		(472)					
OCDD ³		(458)		(460)					
13C-2378-TCDF**		(316)		(318)					
2378-TCDF ⁴		(304)		(306)					
TOTAL-TCDF ⁴		(304)		(306)					
X2378-PeCDF ⁴		(342)		(340)					
TOTAL-PeCDF ⁴		(342)		(340)					
XX2378-HxCDF ⁵		(376)		(374)					
TOTAL-HxCDF ⁵		(376)		(374)					
13C-1234678-HpCDF***		(422)		(420)					
XXX2378-HpCDF ⁵		(410)		(408)					
TOTAL-HpCDF ⁵		(410)		(408)					
OCDF ⁵		(442)		(444)					

- * - Ion used for quantitation
 ** - Quantitation based on 13C-1,2,3,4-TCDD
 *** - Quantitation based on 13C-1,2,3,7,8,9-HxCDD
 1 - Int Std 13C-2,3,7,8-TCDD
 2 - Int Std 13C-1,2,3,6,7,8-HxCDD
 3 - Int Std 13C-OCDD
 4 - Int Std 13C-2,3,7,8-TCDF
 5 - Int Std 13C-1,2,3,4,6,7,8-HpCDF

TEF _____

NOTE: If more than one ratio is required please write ratios directly on ZICP

FORM 7
QUALITY CONTROL REPORT

CASE NO. _____
SITE _____
EPA SAMPLE ID. _____

MATRIX SPIKE/MATRIX SPIKE DUPLICATE RESULTS

COMPOUND	SAMPLE CONC.	SPIKE ADDED MS	MS CONC.	MS %REC	SPIKE ADDED MSD	MSD CONC.	MSD %REC.	%RPD
(units)	()	()	()		()	()		
TCDD								
PeCDD								
HxCDD								
HpCDD								
OCDD								
TCDF								
PeCDD								
HxCDD								
HpCDD								
OCDD								

Sample No.: _____

Analyt(e): _____

[illegible][illegible]

B-13a

DIOXIN RAW SAMPLE DATA

Laboratory: _____

Sample No.: _____

Case/Batch No.: _____

Analyst(s): _____

PCDD Required 358/356 Ratio Window is 0.55-0.75

No. Peaks	Scan No.		Confirm as PCDD Y/N	Quantitated vs C-13 2378-TCDD	C-13 1234-TCDD	Dilution	Conc.
	358/356						
							Total PeCDD:

PCDF Required 342/340 Ratio Window is 0.55-0.75

No. Peaks	Scan No.		Con- firmed as PCDD Y/N	Quantitated vs C-13 237B-TCDD	C-13 1234-TCDD	Dilution	Conc.
	342/340						
						Total PeCDF:	

Sample No. : _____

Analyt(s): _____

No. Peaks	Scan No.		Confirm as PCDD Y/N	Quantitated vs C-13 2378-TCDD	C-13 1234-TCDD	Dilution	Conc.
		392/390					
						Total HxCDD:	

[illegible][illegible]

DIOXIN RAW SAMPLE DATA

Laboratory: _____

Sample No.: _____

Case/Batch No.: _____

Analyst(s): _____

HpCDD Required 426/424 Ratio Window is 0.83-1.12

No. Peaks	Scan No.		Confirm as HpCDD Y/N	Quantitated vs C-13 OCDD	C-13 1234-TCDD	Dilution	Conc.
		426/424					
							Total HpCDD:

HpCDF Required 410/408 Ratio Window is 0.83-1.12

No. Peaks	Scan No.		Confirmed as HpCDF Y/N	Quantitated vs C-13 OCDD	C-13 1234-TCDD	Dilution	Conc.
		410/408					
							Total HpCDF:

B-13d

DIOXIN RAW SAMPLE DATA

Laboratory: _____

Sample No. 1

Case/Batch No.: _____

Analyst(s): _____

OCDD Required 458/460 Ratio Window is 0.75-1.01

No. Peaks	Scan No.		Confirm as OCDD Y/N	Quantitated vs C-13 OCDD	C-13 1234-TCDD	Dilution	Conc.
		458/460					
							Total OCDD:

OCDF Required 442/444 Ratio Window is 0.75-1.01

No. Peaks	Scan No.		Confirmed as OCDD Y/N	Quantitated vs C-13 OCDD	C-13 1234-TCDD	Dilution	Conc.
		442/444					
							Total OCDF:

REC'D

10-31-90

FB

CIBA-GEIGY

INORGANIC REGION I WORKSHEETS

RE-EDITED FOR APPENDIX IX

CONSTITUENTS

Prepared by:

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Radian Corporation
8501 MO-PAC Blvd.
Austin, Texas 78720

CIBA-GEIGY
INORGANIC REGION I WORKSHEET

✓

Background: The term hazardous constituent used in the Solid Waste Disposal Act Section 3004(u) means constituents found in Appendix VIII of 40CFR part 261. EPA also defines hazardous constituents as those constituents identified in Appendix IX of 40CFR part 264. Appendix IX constituents generally constitutes a subset of Appendix VIII which are particularly suitable for ground water analyses. However, it also includes additional constituents not found in Appendix VIII, but commonly addressed in ground water analyses conducted as part of Superfund cleanups.

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analyzed for

sketch

sketch

In general, where very little is known of waste characteristics, and where there is a potential for a wide spectrum of wastes to have been released, only then is an owner/operator required to perform an extensive routine analysis for a broader spectrum of waste such as an Appendix IX analysis.

Radian Corporation of Austin, Texas, has been sub-contracted by WCC to analyze the 232 hazardous constituents in Appendix IX and will be utilizing the following SW-846 procedures listed in Table I. As such, the enclosed Inorganic Region I Data Validation Worksheets have been modified accordingly for each fraction to conform to the QA/QC criteria of each SW-846 test methods in Table I.

TABLE I
SELECTED ANALYTICAL METHODS FOR
INORGANIC APPENDIX IX ANALYSES

SW-846 Method	General Category/ Analyte	Technique	Number of Analytes Measured
6010	Metals*	ICP	11
7041	Antimony	GFAA	1
7060	Arsenic	GFAA	1
7421	Lead	GFAA	1
7470	Mercury	CVAA	1
7740	Selenium	GFAA	1
7841	Thallium	GFAA	1
8080	Organochlorine Pesticides and PCBs	GC/ECD	28
8140	Organophosphorus Pesticides	GC/FPD	9
8150	Herbicides	GC/ECD	4
8240	Volatile Organics	GC/MS	* 54
8270	Semivolatile Organics	GC/MS	** 111
8280	Dioxins and Furans	GC/MS	7
9012	Cyanide	Colorimetric	1
9030	Sulfide	Titrimetric	1
TOTAL			238 19

ICP - Inductively Coupled Plasma Spectrometry (all other metals) *antimony*
 GFAA - Graphite Furnace Atomic Absorption Spectrometry (*arsenic, lead, selenium*
 GC/ECD - Gas Chromatography/Electron Capture Detection *normally only and thallium*
 GC/FPD - Gas Chromatography/Flame Photometric Detection
 GC/MS - Gas Chromatography/Mass Spectrometry

CVAA - Cold Vapor Atomic Absorption Spectroscopy (*mercury analyses only*)
 * This number includes three analytes (1,4-Dioxane, isobutanol, methacrylonitrile) that will be analyzed by Method 8240 Direct Injection.
 ** This number includes Appendix IX analytes, however, this number will increase based on site specific compounds that will be analyzed by Method 8270. Sym-Trinitrobenzene will need to be analyzed as a tentatively identified compound due to the unavailability of standard.

* Metals include: *antimony, arsenic, barium, beryllium, cadmium, chromium, cobalt, copper, lead, mercury, nickel, selenium, silver, thallium, tin, vanadium and zinc*

Inorganic Metals

Summary Table

as with Organic VOA-Waters

(to be developed)

REGION I REVIEW OF INORGANIC
~~CONTRACT LABORATORY DATA PACKAGE~~
SW-846 (3rd Edition)

The hardcopied (laboratory name) _____ data package received at Region I has been reviewed and the quality assurance and performance data summarized. The data review included:

~~Case No.:~~ ← ~~SAS No.:~~ { Sampling Date(s):
 SDG. No.: Matrix : { Shipping Date(s):
 No. of Samples: { Date Rec'd by Lab:
 Sample Identifiers &
 ~~Traffic Report Nos.:~~
 ← Trip Blank No.:
 ← Equipment Blank No.:
 Field Dup Nos.:

SW-846 (3rd Edition)

~~SOW No. _____~~ requires that specific analytical work be done, and that associated reports be provided by the laboratory to the Regions, EMSL, LV, and SMO. The general criteria used to determine the performance were based on an examination of:

- | | |
|---------------------------------|------------------------------|
| -Data Completeness | -Field Duplicates |
| -Holding Times | -Lab Control Sample Results |
| -Calibrations | -Furnace AA Results |
| -Blanks | -ICP Serial Dilution Results |
| -ICP Interference Check Results | -Detection Limit Results |
| -Matrix Spike Recoveries | -Sample Quantitation |
| -Laboratory Duplicates | |

Overall Comments:

Definitions and Qualifiers:

- A - Acceptable data.
 J - Approximate data due to quality control criteria.
 R - Reject data due to quality control criteria
 U - Analyte not detected

Reviewer:

Date:

REGION I
Data Review Worksheets

I. DATA COMPLETENESS

MISSING INFORMATION

DATE LAB CONTACTED

DATE REC'D

REGION I
Data Review Worksheets

II. HOLDING TIMES

Complete table for all samples and circle the analysis date for samples not within criteria.

SAMPLE ID	DATE SAMPLED	HG DATE ANALYSIS	CYANIDE DATE ANALYSIS	OTHERS DATE ANALYSIS	pH	ACTION
--------------	-----------------	------------------------	-----------------------------	----------------------------	----	--------

METALS - 180 DAYS FROM SAMPLE COLLECTION
MERCURY - 28 DAYS FROM SAMPLE COLLECTION
CYANIDE - 14 DAYS FROM SAMPLE COLLECTION

ACTION:

1. If holding times are exceeded all positive results are estimated (J) and non-detects are estimated (UJ).
2. If holding times are grossly exceeded, the reviewer may determine that non-detects are unusable (R).

REGION I
Data Review Worksheets

III A. INSTRUMENT CALIBRATION (Section 1)

1. Recovery Criteria

List the analytes which did not meet the percent recovery (%R) criteria for Initial or Continuing Calibration.

<u>DATE</u>	<u>ICV/CCV#</u>	<u>ANALYTE</u>	<u>%R</u>	<u>ACTION</u>	<u>SAMPLES AFFECTED</u>
-------------	-----------------	----------------	-----------	---------------	-------------------------

ACTIONS:

If any analyte does not meet the %R criteria follow the actions stated below:

For Positive Results:

	<u>Accept</u>	<u>Estimate (J)</u>	<u>Reject (R)</u>
Metals	90-110%R	75-89%R, 111-125%R	<75%R, >125%R
Mercury	80-120%R	65-79%R, 121-135%R	<65%R, >135%R
Cyanide	85-115%R	70-84%R, 116-130%R	<70%R, >130%R

For Non-detected Results:

	<u>Accept</u>	<u>Estimate (UJ)</u>	<u>Reject (R)</u>
Metals	90-125%R	75-89%R	<75%R, >125%R
Mercury	80-135%R	65-79%R	<65%R, >135%R
Cyanide	85-130%R	70-84%R	<70%R, >130%R

III B. INSTRUMENT CALIBRATION (Section 2)

2. Analytical Sequence

(3rd Edition)

- A. Did the laboratory use the proper number of standards for calibration as described in ~~the~~ ~~SOW~~ SW-846? *There is no difference on the proper number of standards between the CLP-SOW and SW-846.* Yes or No
- B. Were calibrations performed at the beginning of each analysis? *day (or every 8 hours); whichever is more frequent.* Yes or No
- C. Were calibration standards analyzed at the beginning of sample analysis and at a minimum frequency of ten percent? *midpoint* ~~or every two hours during analysis, whichever is more frequent?~~ Yes or No
- D. Were the correlation coefficients for the calibration curves for AA, Hg, and CN ≤ 0.995 ? Yes or No
- E. ~~Was a standard at 2xCRDL analyzed for all ICP analyses?~~ ~~Yes or No~~

If No,

The data may be affected. Use professional judgement to determine the severity of the effect and qualify the data accordingly. Discuss any actions below and list the samples affected.

IV A. BLANK ANALYSIS RESULTS (Sections 1-3)

List the blank contamination in Sections 1 & 2 below. A separate worksheet should be used for soil and water blanks.

MATRIX: _____

1. Laboratory Blanks

<u>DATE</u>	<u>ICB/CCB#</u>	<u>PREP BL</u>	<u>ANALYTE</u>	<u>CONC./UNITS</u>
-------------	-----------------	----------------	----------------	--------------------

2. Equipment/Trip Blanks

<u>DATE</u>	<u>EQUIP BL#</u>	<u>ANALYTE</u>	<u>CONC./UNITS</u>
-------------	------------------	----------------	--------------------

3. Frequency Requirements

- insert ①*
- Revised* A. Was a preparation blank analyzed for each matrix ~~for every 20 samples and for each digestion batch?~~ (sample delivery group)? *observed* Yes or No
- B. Was a calibration blank run every 10 samples or every 2 hours whichever is more frequent? *within a calibration* Yes or No
- Curve with a minimum of three standards*
- If No, *Yes for the first* *at least one standard is at the point - range or near*

The data may be affected. Use professional judgement to determine the severity of the effect and qualify the data accordingly. Discuss any actions below, and list the samples affected.

If 20 or more samples per day were analyzed or if 50 or more samples were analyzed, the working standard curve should have an additional standard at or near the midrange for every 10 samples.

Any anomalies encountered?

inset ①

- A. Was a preparation blank carried through the entire analytical process for each matrix type or group of 20 samples, whichever is more frequent? Yes or No
- B. Was a reagent blank with a minimum of three standards run daily? Yes or No
- C. If 20 or more samples were run, was a reagent blank analyzed along with a mid-range standard at a frequency of every 10 samples? Yes or No

IV B. BLANK ANALYSIS RESULTS (Section 4)

4. Blank Actions

The Action Levels for any analyte is equal to five times the highest concentration of that element's contamination in any blank. The action level for samples which have been concentrated or diluted should be multiplied by the concentration/dilution factor. No positive sample result should be reported unless the concentration of the analyte in the sample exceeds the Action Level (AL). Specific actions are as follows:

1. When the concentration is greater than the IDL, but less than the Action Level, report the sample concentration detected with a U.
2. When the sample concentration is greater than the Action Level, report the sample concentration unqualified.

MATRIX: _____

MATRIX: _____

<u>ELEMENT</u>	<u>MAX. CONC./</u> <u>UNITS</u>	<u>AL/</u> <u>UNITS</u>
----------------	------------------------------------	----------------------------

<u>ELEMENT</u>	<u>MAX. CONC./</u> <u>UNITS</u>	<u>AL/</u> <u>UNITS</u>
----------------	------------------------------------	----------------------------

NOTE: Blanks analyzed during a soil case must be converted to mg/kg in order to compare them with the sample results.

$$\text{Conc. in ug/L} \times \frac{\text{Volume diluted to } (200\text{ml})}{\text{Weight digested (1 gram)}} \times \frac{1\text{L}}{1000\text{ ml}} \times \frac{1000\text{gm}}{1\text{ kg}} \times \frac{1\text{mg}}{1000\text{ug}} = \text{mg/kg}$$

Multiplying this result by 5 to arrive at the action level gives a final result in mg/kg which can then be compared to sample results.

The SW-846 (3rd Edition) requirement is that the calibration blank be within three (3) standard deviation window of the mean blank value. As such, gross blank contamination warrants the data validator to contact the laboratory to verify this

ms90-210

Page 7 of 18

was performed. List all anomalies in the Inorganic Regional Data Assessment.

REGION I
Data Review Worksheets

V A. ICP INTERFERENCE CHECK SAMPLE (Sections 1 & 2)

1. Recovery Criteria

List any elements in the ICS AB solution which did not meet the criteria for %R. SW-846 (3rd Edition) does warrant a $\pm 20\%$ window of the true value. As such, all Action Percent Recoveries are considered acceptable.

<u>DATE</u>	<u>ELEMENT</u>	<u>%R</u>	<u>ACTION</u>	<u>SAMPLES AFFECTED</u>
-------------	----------------	-----------	---------------	-------------------------

ACTIONS:

If an element does not meet the %R criteria, follow the actions stated below:

	<50%	<u>PERCENT RECOVERY</u>		>120%
		50-79%		
Positive Sample Results	R	J		J
Non-detected Sample Results	R	UJ		A

2. Frequency Requirements

Were Interference QC samples run at the beginning and end of each ~~sample~~ analysis run or a minimum of twice per 8 hour working shift, whichever is more frequent? Yes or No

batch

If no,

The data may be affected. Use professional judgement to determine the severity of the effect and qualify the data accordingly. Discuss any actions below and list the samples affected.

REGION I
Data Review Worksheets

V B. ICP INTERFERENCE CHECK SAMPLE (Section 3)

- Report the concentration of any elements detected in the ICS A solution > 2xIDL that should not be present.

<u>ELEMENT</u>	<u>CONC. DETECTED</u> <u>IN THE ICS</u>	<u>CONC. OF INTERFERENTS</u> <u>IN THE ICS</u>			
		<u>AL</u>	<u>CA</u>	<u>FE</u>	<u>MG</u>

Estimate the concentration produced by the interfering element in all affected samples. See guidelines for examples. List the samples affected by interferences below:

<u>SAMPLE</u> <u>AFFECTED</u>	<u>ELEMENT</u> <u>AFFECTED</u>	<u>SAMPLE</u> <u>CONC.</u> <u>(ug/L)</u>	<u>SAMPLE INTERFERENT</u> <u>CONC.</u>				<u>ESTIMATED</u> <u>INTERF.</u> <u>(ug/L)</u>
			<u>AL</u>	<u>CA</u>	<u>FE</u>	<u>MG</u>	

ACTIONS:

- In general, the sample data can be accepted without qualification if the sample concentrations of Al, Ca, Fe, and Mg are less than 50% of their respective levels in the ICS solution.
- Estimate (J) positive results for affected elements for samples with levels of interferents 50% or more of that in the ICS solution.
- Reject (R) positive results if the reported concentration is due entirely to the interfering element.
- Estimate (UJ) non-detected results for which false negatives are suspect.

Give explanations for any actions taken below:

REGION I
Data Review Worksheets

VI. MATRIX SPIKE

TR # _____

MATRIX: _____

1. Recovery Criteria

List the percent recoveries for analytes which did not meet the required criteria.

S - amount of spike added
SSR - spikes sample request
SR - sample result

Analyte	SSR	SR	S	%R	Action
---------	-----	----	---	----	--------

Matrix Spike Actions apply to all samples of the same matrix.

ACTIONS:

1. If the sample concentration exceeds the spike concentration by a factor of 4 or more, no action is taken.
2. If any analyte does not meet the %R criteria follow the actions stated below:

	PERCENT RECOVERY		
	<30%	30%-74%	>125%
Positive Sample Results	J	J	J
Non-detected Results	R	UJ	J

2. Frequency Criteria

A. Was a matrix spike prepared at the required frequency?
Yes or No

B. Was a post digestion spike analyzed for elements that did not meet required criteria for matrix spike recovery?
Yes or No

A separate worksheet should be used for each matrix spike pair.

REGION I
Data Review Worksheets

VII. LABORATORY DUPLICATES

List the concentrations of any analyte not meeting the criteria for duplicate precision. For soil duplicates, calculate the ~~CRDL~~ ^{PQL} in mg/kg using the sample weight, volume and percent solids data for the sample. Indicate what criteria was used to evaluate precision by circling either the RPD or ~~CRDL~~ for each element.

PQL

PQL* ~~NOT~~ ~~CRDL~~ ~~NOT PQL~~ MATRIX: _____

Element	water ug/L	soil mg/kg	Sample #	Duplicate #	RPD**	Action
Aluminum	200					
Antimony	30 50					
Arsenic	2 10					
Barium	10 200					
Beryllium	2 5					
Cadmium	5 5					
Calcium	5000					
Chromium	10					
Cobalt	20 50					
Copper	20 25					
Iron	100					
Lead	2 5					
Magnesium	5000					
Manganese	15					
Mercury	0.2					
Nickel	20 40					
Potassium	5000					
Selenium	2 5					
Silver	10 10					
Sodium	5000					
Thallium	2 10					
Vanadium	20 50					
Zinc	20					
Cyanide	10					

50/100

Laboratory Duplicate Actions should be applied to all other samples of the same matrix type.

ACTIONS:

- Estimate (J) positive results for elements which have an RPD >20% for waters and >35% for soils.
- If sample results are less than 5x the ~~CRDL~~ ^{PQL}, estimate (J) ~~NOT~~ ^{PQL} positive results for elements whose absolute difference is >~~CRDL~~ ^{PQL}, (2x~~CRDL~~ for soils). If both samples are non-detected, the RPD is not calculated (NC).

* No detection limits are required in SW-846. All ~~MDL~~ ^{PQL} are of a recommended nature. As such, all CLP-CRDLs will be submitted as MDL for reporting purposes. Page 11 of 18

** Mean RPD of $\pm 20\%$ ^{± 2 standard deviations} of the 19 or 25 runs (whichever is higher) for all analytes greater than ~~MDL~~ ^{PQL} as warranted based on SW-846 (3rd Edition) protocols.

REGION I
Data Review Worksheets

VIII. FIELD DUPLICATES

List the concentrations of all analytes in the field duplicate pair. For soil duplicates, calculate the CRDL in mg/kg using the sample weight, volume and percent solids data for the sample. Indicate what criteria was used to evaluate the precision by circling either the RPD or CRDL for each element.

PQL
MDE

MATRIX: _____

Element	CRDL		Sample #	Duplicate #	RPD **	Action
	water ug/L	soil mg/kg				
Aluminum	200					
Antimony	30 50					
Arsenic	2 10					
Barium	10 200					
Beryllium	5					
Cadmium	5					
Calcium	5000					
Chromium	10					
Cobalt	20 50					
Copper	20 25					
Iron	100					
Lead	2 5					
Magnesium	5000					
Manganese	15					
Mercury	0.2					
Nickel	20 40					
Potassium	5000					
Selenium	2 5					
Silver	10 10					
Sodium	5000					
Thallium	2 10					
Vanadium	20 50					
Zinc	20					
Cyanide	10					

5-10-1 (circled) points to Chromium

5-10-1 (circled) points to Vanadium

TIN points to Cyanide

100 (circled) points to Sodium

Field duplicate actions should be applied to all other samples of the same matrix type.

ACTIONS:

1. Estimate (J) positive results for elements which have an RPD >30% for waters and >50% for soils.
2. If sample results are less than 5x the CRDL, estimate (J) positive results and (UJ) nondetected results for elements whose absolute difference is >2xCRDL, (4xCRDL for soils). If both samples are non-detected, the RPD is not calculated (NC).

* No detection limits are required in SW-846. All *PQL* *MDE* are of a recommended nature. As such, all CLP CRDLs will be substituted as *PQL* *MDE* for reporting purposes. Page 12 of 18

** Mean RPD of $\pm 20\%$ (whichever is higher) for all analytes generated or recommended based on SW-846 (3rd Edition) protocols.

IX. LABORATORY CONTROL SAMPLE

1. Aqueous LCS

List any LCS recoveries not within the 80-120% criteria and the samples affected.

<u>DATE</u>	<u>ELEMENT</u>	<u>%R</u>	<u>ACTION</u>	<u>SAMPLES AFFECTED</u>
-------------	----------------	-----------	---------------	-------------------------

Note: The SW-846 (3rd Edition) LCS recovery window is $\pm 20\%$. The current 80-120% is acceptable.

2. Solid LCS

List ^{manufacturers} any analytes that were not within the control windows set by the EPA for the solid LCS sample. ~~The 80-120% criteria is not used to evaluate solid LCS results.~~ *Lot specifications are available on request from Radco®*

<u>ELEMENT</u>	<u>LCS CONC.</u>	<u>CONTROL WINDOWS</u>	<u>ACTION</u>	<u>SAMPLES AFFECTED</u>
----------------	------------------	------------------------	---------------	-------------------------

ACTIONS:

<u>AQUEOUS LCS</u>	<u><50%</u>	<u>51-79%</u>	<u>>120%</u>
Positive Results	R	J J	
Non-detected Results	R	UJ A	

<u>SOLID LCS</u>	<u><EPA Control Windows</u>	<u>>EPA Control Windows</u>
Positive Results	J	J
Non-detected Results	UJ	A

3. Frequency Criteria

A. Was an LCS analyzed for every matrix, every digestion batch, and every 20 samples? Yes or No

X A. FURNACE ATOMIC ABSORPTION ANALYSIS

1. Duplicate Precision

~~Duplicate injections and one-point analytical spikes were performed for all samples: duplicate injections agreed within $\pm 20\%$.~~

~~Duplicate injections and/or spikes were not performed for the following samples/elements:~~

do
~~of Duplicate injections did not agree within $\pm 20\%$ for samples/elements~~ *the laboratory must return and report the lowest coefficient of variation as per SW-846 (3rd Edition) protocols*

2. Post Digestion Spike Recoveries

Spike recoveries met the ~~85-115%~~ *75-125* recovery criteria for all samples.

Spike recoveries did not meet the ~~85-115%~~ *75-125* criteria, but did not require MSA for the following samples/elements:

MSA was used to quantitate analytical results when contractually required.

Correlation coefficients >0.995 , accept results.
Correlation coefficients <0.995 for sample numbers/elements:

Method of Standard Addition (MSA) ~~was not performed as during~~ *is (3rd Edition) protocols being used of sampling.*
~~required for samples/elements:~~

Note: CLP requirements and not SW-846 will be used as guidance when applying the actions below.
ACTIONS:

1. Estimate (J) positive results if duplicate injections are outside $\pm 20\%$ RSD or CV.
2. If the sample absorbance is $<50\%$ of post digestion spike absorbance the following actions should be applied:

	PERCENT RECOVERY			
	<u>$<10\%$</u>	<u>$11\%-84\%$</u>		<u>$>115\%$</u>
Positive Sample Results	J or R	J	J	
Non-detected Results	R	UJ	A	

3. Estimate (J) sample results if MSA was required, ~~and not performed.~~

4. Estimate (J) sample results if correlation coefficient was <0.995 .

XI. INDUCTIVELY COUPLED PLASMA (ICP) SERIAL DILUTION ANALYSIS

Serial Dilutions were performed for each matrix and results of the diluted sample analysis agreed within ten percent of the original undiluted analysis, *as per CLP guidelines and not SW-846 (3rd Edition) protocols*
Serial Dilutions were not performed for the following:

Serial Dilutions were performed, but analytical results did not agree within 10% for analyte concentrations greater than 50x the IDL before dilution.

Report all results below that do not meet the required laboratory criteria for ICP serial dilution analysis.

MATRIX: _____

ELEMENT	IDL	50xIDL	SAMPLE RESULT	SERIAL DILUTION	%D	ACTION
Aluminum						
Antimony						
Arsenic						
Barium						
Beryllium						
Cadmium						
Calcium						
<i>5421</i> Chromium						
Cobalt						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Nickel						
Potassium						
Silver						
Sodium						
Vanadium						
Zinc						

Actions apply to all samples of the same matrix.

ACTIONS:

1. Estimate (J) positive results if %D >15.

Note: Sample result must be $\geq 50 \times$ ^{PQL} IDL for calculation by serial dilution; then use $\pm 10\%$ original undiluted value

XII. DETECTION LIMIT RESULTS

1. Instrument Detection Limits

Instrument Detection Limit results were present and found to be less than the Contract Required Detection Limits.

IDLs were not included in the data package on Form XI.

IDLs were present, but the criteria was not met for the following elements: _____

2. Reporting Requirements

Were sample results on Form I reported down to Yes or No the IDL not the CRDL for all analytes?

Were sample results that were analyzed by ICP for Se, Tl, As, or Pb at least 5X IDL. Yes or No

Were sample weights, volumes, and dilutions taken into account when reporting detection limits on Form I. Yes or No

If No,

The reported results may be inaccurate. Make the necessary changes on the data summary tables and request that the laboratory resubmit the corrected data.

XIII. SAMPLE QUANTITATION

Sample results fall within the linear range for ICP and within the calibrated range for all other parameters.

Sample results were beyond the linear range/calibration range of the instrument for the following samples/elements:

In the space below, please show a minimum of one sample calculation per method:

ICP

Lab formula: $\frac{\mu\text{g}}{\mu\text{l}} \times \frac{100\text{ml}}{\text{g}} = \mu\text{g/g} (\text{mg/kg})$

FURNACE

Water $\frac{\mu\text{g}}{\text{L}} \times \frac{100\text{ml}}{100\text{ml}} = \mu\text{g/L}$

MERCURY

CYANIDE

For soil samples, the following equation may be necessary to convert raw data values (usually reported in $\mu\text{g/L}$) to actual sample concentrations (mg/kg):

The lab is required to use 1 gram sample (wet weight) to ¹⁰⁰~~200~~ ml.

Wet weight concentration =

^{SOLID}
digest conc. in $\frac{\mu\text{g}}{\text{L}} \times \frac{100}{1\text{ gm}} \times \frac{1\text{L}}{1000\text{ ml}} \times \frac{1000\text{gm}}{1\text{kg}} \times \frac{1\text{mg}}{1000\mu\text{g}} = \frac{\text{mg}}{\text{kg}}$

In addition the sample results are converted to dry weight using the percent solids calculations:

$\frac{\text{Wet weight conc.}}{\% \text{solids}} \times 100 = \text{final concentration, dry weight (mg/kg)}$

INORGANIC REGIONAL DATA ASSESSMENT

*Reference*CASE NO. _____
LABORATORY _____SITE _____
NO. OF SAMPLES/
MATRIX _____

SDG # _____

REVIEWER (IF NOT ESD) _____

SW 876 SOW # _____

REVIEWER'S NAME _____

DPO: ACTION _____ FYI _____

COMPLETION DATE _____

DATA ASSESSMENT SUMMARY

	ICP	AA	Hg	CYANIDE
1. HOLDING TIMES	_____	_____	_____	_____
2. CALIBRATIONS	_____	_____	_____	_____
3. BLANKS	_____	_____	_____	_____
4. ICS	_____	_____	_____	_____
5. LCS	_____	_____	_____	_____
6. DUPLICATE ANALYSIS	_____	_____	_____	_____
7. MATRIX SPIKE	_____	_____	_____	_____
8. MSA (<i>not performed</i>)	_____	_____	_____	_____
9. SERIAL DILUTION	_____	_____	_____	_____
10. SAMPLE VERIFICATION	_____	_____	_____	_____
11. OTHER QC	_____	_____	_____	_____
12. OVERALL ASSESSMENT	_____	_____	_____	_____

O = Data had no problems/or qualified due to minor problems.

M = Data qualified due to major problems.

Z = Data unacceptable.

X = Problems, but do not affect data.

ACTION ITEMS: _____

AREAS OF CONCERN: _____

NOTABLE PERFORMANCE: _____